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(21) International Application Number: PCT/US99/24971 (22) International Filing Date: 22 October 1999 (22.10.99) (30) Priority Data: 09/176,814 22 October 1998 (22.10.98) US (71) Applicants: MATHESON TRI GAS, INC. [US/US]; 1861 Lefthand Circle, Longmont, CO 80501 (US). NIPPON SANSO CORPORATION [JP/JP]; 1-16-7 Nishishinbashi, Minato-ku, Tokyo 105-8442 (JP). (72) Inventor: WATANABE, Tadaharu; 2875 Shadow Creek Drive #B202, Boulder, CO 80303 (US). (74) Agents: MORRIS, Francis, E. et al.; Pennie & Edmonds LLP, 1155 Avenue of the Americas, New York, NY 10036 (US).		(81) Designated States: CA, CN, JP, KR, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: SCAVENGER FOR REDUCING IMPURITIES IN A GAS AND METHODS AND APPARATUSES FOR MAKING AND USING SAME (57) Abstract Described is a scavenger for reducing the concentration of an impurity in a gas, comprising an inorganic support (20) having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof wherein x is one, two or three, and y is zero, one, two, three, four, five or six. Also described are methods and apparatuses for making and for using the scavenger.		

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SCAVENGER FOR REDUCING IMPURITIES IN A GAS AND METHODS AND APPARATUSES FOR MAKING AND USING SAME

5 BACKGROUND OF THE INVENTION

Field of the Invention

The invention relates generally to the removal of impurities contained in gas
10 streams. More specifically, the invention relates to the removal of oxidants and Lewis acids from inert and hydride gases typically used in semiconductor processing.

Description of Related Art

15 The provision of high purity gas is important in the semiconductor industry. The growth of high quality semiconductor materials by chemical vapor deposition or other vapor-based technology depends upon the use of high purity inert and hydride gases. Impurities in these gases, such as water, oxygen, carbon dioxide, carbon monoxide and other oxidants, and Lewis acids such as aluminum, boron and zinc-related species, can
20 result in reduced yields of such materials, due to defects in crystalline structure and epitaxy, and reduced electrical properties and performance. Tolerable impurity levels are currently on the order of 1ppm for hydride gases and as low as a few ppb for inert gases.

Several technologies have been proposed to reduce these impurities to acceptable concentration levels including physical and chemical adsorption. Physical adsorption
25 methods include the use of zeolites, alumina, silica, activated carbon and similar performing compounds that physically adsorb the impurity. Generally, however, these processes do not provide sufficient removal efficiencies. Chemical adsorption methods include, for example, the use of getter compounds, nickel, titanium and copper which adsorb and chemically react with the impurity. These methods are capable of providing sufficient removal efficiencies,
30 but they are complex and relatively expensive due to the heat required to activate the reactant. Other chemical adsorption methods use alkaline and alkaline earth metals, and their respective alkyl compounds, on high surface area supports. However, these processes may release hydrocarbons or metals into the gas stream, thereby causing further contamination of the gas. Even very low amounts of hydrocarbon contamination may result

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in the incorporation of carbon in a growing film due to decomposition of the hydrocarbon during the chemical vapor deposition process.

Some of the different types of compounds that are used to chemically adsorb certain impurities from inert gases are specifically describes in several United States Patents. Tom (United States Patent No. 4,716,181) describes a polymer scavenger comprising a metallated macroreticulate polymer functionalized by arsine or phosphine and used to remove impurities that can be metallated, such as water, oxygen and carbon dioxide from gases such as arsine and phosphine. It is noted that this scavenger is hydrocarbon and metal based and, therefore, may further contaminant the gas stream. In addition, the generation of this scavenger requires at least two reaction steps. Tom (United States Patent No. 4,781,900) describes a scavenger comprising a carbanion, preferably an organometallic compound, or a carbanion that has been reacted with arsine, phosphine or ammonia, that is used to remove impurities such as Lewis acids and oxidants from inert gases. It is noted that this scavenger is also hydrocarbon and metal based.

Tom (United States Patent No. 4,950,419) describes a scavenger comprising a pyrolyzed alkyl metal compound selected from Group IA, IIA and/or IIIA of the Periodic Table that is used to remove impurities such as Lewis acids and oxidants from inert gases. Tom (United States Patent No. 5,531,971) describes a scavenger comprising a pyrolyzed metal selected from Group IA of the Periodic Table that is used to remove water, oxygen, Lewis acids and oxidants from certain inert and hydride gases. Similarly, both of these scavengers are either hydrocarbon and/or metal based and, therefore, may further contaminant the gas stream.

Imai et. al. (Imai et. al., "Monosilane Adsorption on Porous Alumina," J. Electrochem. Soc., 142(9), September, 1995) describe the use of alumina and silica to chemically adsorb monosilane (SiH_4), an impurity in hydride gases, such as AsH_3 and PH_3 , and in waste gases from semiconductor processing. The alumina showed a capacity of approximately 4.5×10^{-4} moles of monosilane per gram of support at a temperature of approximately 100 degrees Celsius. However, Imai et. al. does not specifically address the removal from hydride gases of other impurities of interest in semiconductor processing such as water, oxygen, carbon dioxide, carbon monoxide and other oxidants, and Lewis acids such as aluminum, boron and zinc-related species.

Based on the foregoing there is a need for a simple method for reducing the concentration of impurities in gases, thereby producing a high purity gas suitable for use in semiconductor processing, without the risk of further contaminating the gas.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a scavenger for reducing the concentration of an impurity in a gas, comprising an inorganic support and
5 having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof wherein x is one, two or three, and y is zero, one, two, three, four, five or six.

Another object of the present invention provides a method and apparatus for making a scavenger for reducing the concentration of an impurity in a gas. The method comprises
10 the step of contacting a hydride gas selected from the group consisting of B_2H_6 , SiH_4 , Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof, with an inorganic support.

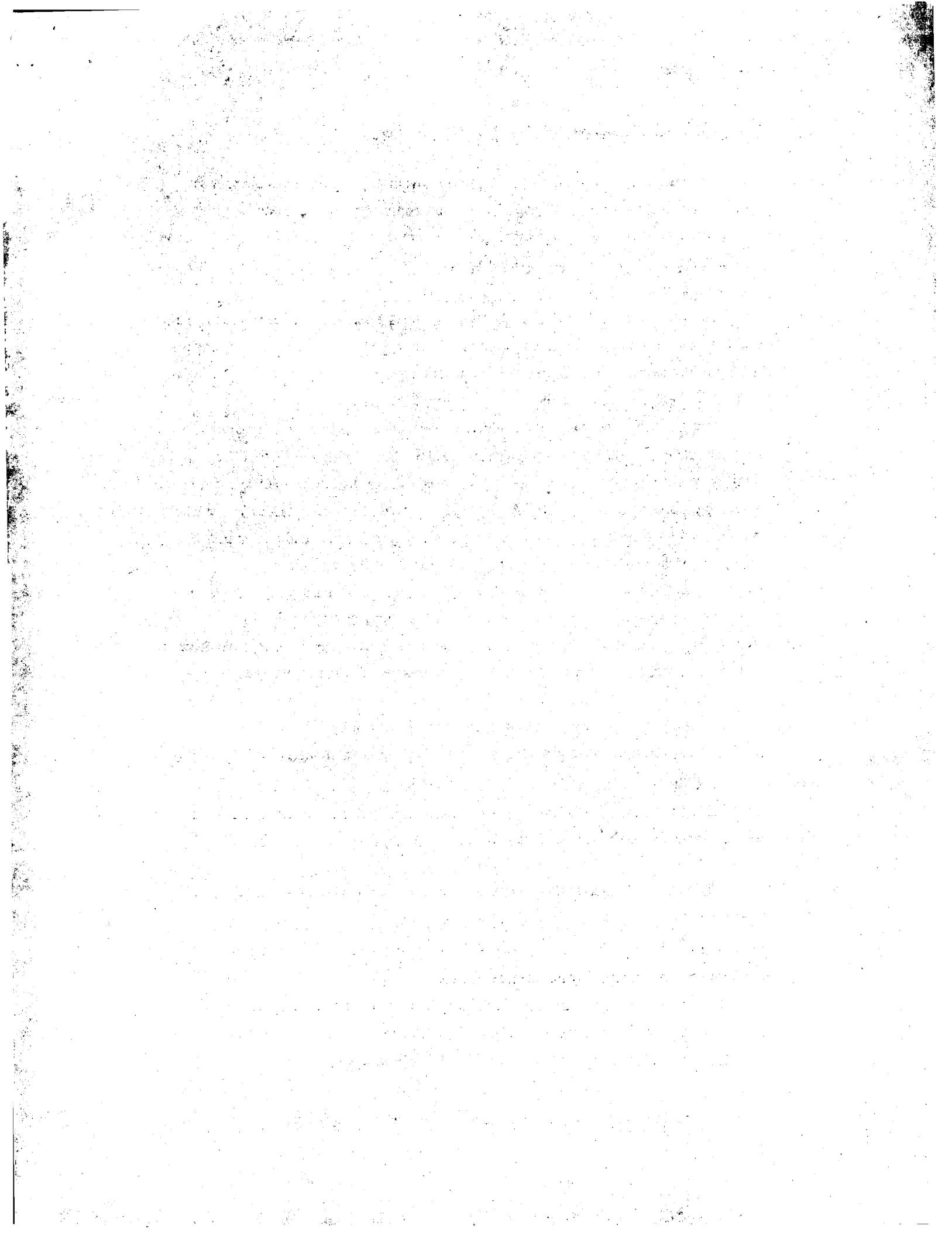
Yet another object of the present invention provides a method and apparatus for using the scavenger. The method comprises the step of contacting a gas containing an impurity with an inorganic support having disposed thereon an active agent selected from
15 the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is an integer selected from the group consisting of one, two and three, and y is an integer selected from the group consisting of one, two, three, four, five and six.

Overall, the scavenger and the method for using it provide a simple process for reducing the concentration of impurities such as water, oxygen, carbon dioxide and other
20 oxidants, and Lewis acids such as aluminum, boron and zinc-related species, in hydride and inert gases, thereby producing a purified gas. The purified hydride or inert gas is particularly suited for use in the semiconductor processing industry, since it has not been further contaminated by the purification process itself, for example, by the addition of metals or hydrocarbons to the gas. Furthermore, the process for making the scavenger is
25 relatively simple.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic view of a process for making the scavenger of the present
30 invention.

FIG. 2 is a schematic view of a process for reducing the concentration of an impurity in a gas stream using the scavenger of the present invention.



DETAILED DESCRIPTION OF THE INVENTION

In general, the scavenger made according to the present invention is useful for reducing the concentration of certain impurities in a gas. The scavenger itself comprises an active agent disposed on the surface of an inorganic support. One benefit of the active agent is that it bonds with sufficient strength to the inorganic support but in a manner that still allows for strong bonding with the adsorbed impurity. Therefore, upon contacting a gas containing certain impurities, the scavenger reacts rapidly and bonds essentially irreversibly with the impurities of interest. Furthermore, since the support for the active agent is inorganic, the scavenger according to the present invention does not release hydrocarbons into the gas stream, thereby avoiding further contamination of the gas by the purification process itself. The product produced by this process is, therefore, a high purity gas.

The specific composition of the active agent is the equivalent of one or more of the following functional groups: B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is one, two or three, and y is zero, one, two, three, four, five or six. The active agent is generally a hydride functional group, with the exception of the cases in which y is zero. It is preferred that the concentration of the active agent on the inorganic support be approximately 0.00003 to 1.5 moles per gram of support and more preferably 0.0002 to 0.05 moles per gram of support. These ranges are based upon the amount of active agent needed to provide sufficient adsorption of the impurities of interest. Furthermore, the range of 0.0002 to 0.05 moles per gram of support corresponds to a monolayer of functional groups on the surface of the inorganic support.

The inorganic support may be selected from zeolite species, alumina, silica and other similar materials. It is important, however, that the support be inorganic to avoid releasing hydrocarbon compounds, found in other types of supports, into the gas, thereby contaminating the gas. In addition, the inorganic support should be thermally stable at temperatures at which the scavenger will be used to insure maintenance of the original structure of the support and to maintain the specific chemical composition of the active agent. Since use of the scavenger at higher temperature enhances the reaction rate between the scavenger and the impurity, the inorganic support should be thermally stable at temperatures up to approximately 150 degrees Celsius and more preferably up to temperatures of approximately 300 degrees Celsius.

The surface area and pore size of the inorganic support are also important as they affect both the production of the scavenger and its ultimate use to remove gaseous impurities. Typically, inorganic supports with high surface areas tend to have small

diameter pores. While higher surface area provides for more contact between the gas impurity and the scavenger, the small diameter pores may actually impede the diffusion of the impurity into the scavenger. However, inorganic supports with larger diameter pores (e.g., greater than 1000 angstroms) typically have inadequate surface area for contacting the scavenger and the gaseous impurities. Therefore, the preferred surface area of the inorganic support is approximately 100 to 900 square meters per gram and more preferably approximately 300 to 700 square meters per gram with pore sizes of approximately 3 to 1000 angstroms and more preferably 200 to 300 angstroms.

The process for making the scavenger involves generating an active agent on an inorganic support by contacting a hydride gas with an inorganic support under conditions which produce a functional group. Specifically, the hydride gas is adsorbed onto the inorganic support and is converted to the active agent by pyrolysis.

The equipment required to contact the hydride gas and the inorganic support can be any equipment known in the art for contacting a gas and a solid. FIG. 1 shows one embodiment of an apparatus 10 for making the scavenger of the present invention. The inlet gas line 14 delivers a hydride gas to the reactor 19 through the inlet gas port 11. The hydride gas passes through an inorganic support 20, which is held in place by a bed support 13, and is adsorbed onto the inorganic support 20. A heater 16 is used to heat the reactor 19 to maintain the desired temperature in the reactor 19 during adsorption and to provide the heat for pyrolysis. The hydride gas then exits the reactor 19 through the outlet gas port 17.

The reactor 19 can be made of any inert material known in the art capable of holding the inorganic support as a fixed bed and having thermal stability at the desired operating temperature. The peripheral equipment, such as the gas lines 14, 17, the heater 16 and the bed support 13, can be the same as those typically used in the art. Further, it should be appreciated that other apparatuses and methods known in the art for contacting a gas and a solid can also be used.

The steps for making the scavenger of the present invention, using the apparatus of FIG. 1, include first loading the inorganic support into the reactor 19 in a fixed bed arrangement which is supported by the bed support 13. It is preferable to dry the inorganic support 20 before introducing the hydride gas into the reactor 19 to remove any water or other adsorbed species that may be attached to the inorganic support 20. This drying step can be accomplished by heating the inorganic support 20 in a dry environment, for example, by purging with a dry, inert gas such as nitrogen for a given period of time at an elevated temperature. It should be appreciated that other methods known in the art for drying the

inorganic support 20 can be used such as drying prior to placing the inorganic support 20 in the reactor 19.

A given hydride gas, or mixture thereof, is then passed through the reactor 19 in a continuous manner and in contact with the inorganic support 20. The hydride gas, or mixture thereof, can also be mixed with an inert gas to provide better control of the hydride gas flow rate.

The hydride gases that may be used include B_2H_6 , SiH_4 , Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof. Depending upon the process conditions used in making the scavenger, in particular the temperature, the use of a specific hydride gas or mixture will produce a corresponding active agent or functional group on the inorganic support. For example, adsorbing SiH_4 hydride gas will result in the inorganic support having Si_xH_y functional groups wherein x is one, two or three and y is zero, one, two, three, four, five or six. More specifically, using SiH_4 gas produces SiH_y functional groups wherein y is zero, one, two or three; using Si_2H_6 gas produces Si_xH_y functional groups wherein x is one or two and y is zero, one, two, three, four or five; using B_2H_6 gas produces B_xH_y functional groups wherein x is one or two and y is zero, one, two, three, four or five; using PH_3 gas produces PH_y functional groups wherein y is zero, one, two or three; and using AsH_3 gas produces AsH_y functional groups wherein y is zero, one, two or three. It should be appreciated that any hydride gas, or mixture thereof, can be used with any inorganic support.

During the step of contacting the hydride gas with the inorganic support, the reactor 19 is heated using a heater 16 to maintain a given temperature in the reactor. The temperature is important as it affects both the adsorption of the hydride gas and the pyrolysis of the adsorbed hydride gas which causes conversion to a functional group (i.e., the active agent). The preferred temperature is between approximately 20 and 500 degrees Celsius and more preferably between approximately 100 and 300 degrees Celsius. As shown in FIG. 1, a heater 16, such as heat tape or a steam jacket, may be used to heat the reactor 19 to maintain the desired temperature. Other methods of directly or indirectly heating a reactor to maintain a given temperature may be used and are well known in the art.

These preferred temperature ranges are based upon competing factors. Although some adsorption of the hydride gas occurs at room temperature, it is preferred that the adsorption occur at higher temperatures to allow for concurrent pyrolysis of the adsorbed gas to convert it to its corresponding functional group. Higher temperatures are also desirable as they favor homogeneous adsorption of the hydride gas which results in a higher concentration of adsorbed gas on the inorganic support. Specifically, homogeneous "end-on" alignment of the adsorbed species results in a higher concentration of adsorbed species

per unit of surface area. For example, end-on alignment of B_2H_6 means that the axis of the B-B bond is normal to the surface of the support, as opposed to "side-on" alignment where the B-B bond is parallel to the surface of the support. Therefore, each adsorbed molecule occupies less surface area, and if a majority of the adsorbed molecules are aligned in a similar manner, the surface area will contain a higher concentration of adsorbed molecules.

Temperatures above 500 degrees Celsius, however, can result in decomposition of the adsorbed hydride gas to its elemental state rather than a conversion of adsorbed hydride gas to its corresponding functional group. Furthermore, lower temperatures produce hydrogen-rich hydride functional groups which are more reactive with the gaseous impurities of interest than hydrogen-poor hydride functional groups produced at higher temperatures. Lastly, temperatures in the range of 100 to 300 degrees Celsius have been found to provide for more control over the adsorption rate of the hydride gas and, therefore, more control over the composition of the scavenger, particularly where combinations of functional groups are desired.

The duration of contact between the hydride gas and the inorganic support 20, given a particular operating temperature and surface area of the inorganic support 20, is that which provides the desired concentration of functional groups on the inorganic support 20. The desired concentration is preferably that at which the inorganic support 20 is saturated by adsorbed hydride gas. One method for determining the concentration of the functional groups on the inorganic support 20 is by sampling the inorganic support 20 and determining the concentration of the active agent on the inorganic support 20. This information can then be used to either increase or decrease the duration of contact. Analysis of the concentration of active agent on the inorganic support 20 can be accomplished by x-ray fluorescence spectroscopy, electron probe micro analysis, Auger electron spectroscopy or electron spectroscopy for chemical analysis. Alternatively, the mass transfer zone in the inorganic support may be measured using thermocouples placed throughout the bed of inorganic support 20 in the reactor 19. Because the adsorption reaction is exothermic, the increase in temperature within the inorganic support 20 can be followed which corresponds to movement of the mass transfer zone. Once the mass transfer zone reaches the end of the bed the capacity of the inorganic support for hydride gas has been reached. Preferably, the duration of contact can be longer than the time for the mass transfer zone to reach the end of the bed to ensure that the inorganic support has been saturated with hydride gas.

Once the desired concentration of active agent on the inorganic support 20 has been obtained, or the appropriate duration of contact has been completed, the inorganic support 20 now containing the active agent, and more appropriately referred to as a scavenger, can

alternatively be purged with an inert gas such as nitrogen. This purge acts to remove any physically adsorbed hydride gas. The scavenger can then be removed from the reactor 19 and used immediately to remove the impurities of interest from a gas, or it can be stored for periods of approximately one year.

- 5 In use, a scavenger prepared according to the present invention is particularly suited to adsorb impurities such as water, oxygen, carbon dioxide and other oxidants, and Lewis acids such as aluminum, boron and zinc-related species from gases such as hydrogen, nitrogen, noble gases (e.g., helium, neon, argon, krypton and xenon), diboran, hydride gases from Groups IVA and VIA of the Periodic Table (e.g., arsine, phosphine, silane, germanium, hydrogen selenide and hydrogen telluride) and ammonia. Furthermore, since
10 the scavenger according to the present invention has an inorganic support, its use will not release hydrocarbons into the gas stream, thereby providing a high purity gas which is suitable for use in semiconductor manufacturing.

- Specifically, a gas containing an impurity is contacted with the scavenger prepared
15 according to the present invention. FIG. 2 shows one embodiment of an apparatus 100 for using the scavenger in accordance with the present invention. The inlet gas line 105 delivers a gas containing one or more impurities to through the inlet gas port 101 to reactor 109 which can be made from any suitable material, such as stainless steel. The gas passes through the reactor 109 and contacts the scavenger 104 which adsorbs the impurities. The
20 scavenger 104 is held in place by a bed support 103. The gas exits the reactor 109 through the outlet gas port 102, and a purified gas is delivered by the outlet gas line 107.

- The actual process steps for contacting the gas with the scavenger are those typical of gas-solid adsorption processes and are well known in the art. It should be appreciated that the method of the present invention will not introduce any hydrocarbons or metals into
25 the purified gas. In addition, it should be appreciated that the scavenger of the present invention can be used to produce a purified gas in cylinders for shipment to another location or the scavenger can be used at the location where the purified gas is used.

- The invention having been described, the following examples are presented to illustrate, rather than to limit the scope of the invention. Three examples illustrate the
30 preparation of three different scavengers according to the present invention, and one example illustrates the results of the use of these scavengers to remove gas impurities according to the present invention.

EXAMPLE 1: Preparation of Si_xH_y Functionalized Silica Scavenger by Si_2H_6
Deposition

5 60 cc of silica gel with a specific surface area of 300 square meters per gram was loaded into a cylindrical reactor having a diameter of 2 cm and a length of 30 cm. The bed height formed by the silica gel was 20 cm. The silica gel was then purged with dry nitrogen for a period of 3 hours at a bed temperature of 150 degrees Celsius. A gas stream of dry nitrogen containing 20% Si_2H_6 was passed through the reactor and silica bed at a flow rate of 1 liter per minute at a bed temperature of 150 degrees Celsius for 10 hours. The silica, now supporting an active agent of the form Si_xH_y , wherein x is one or two and y is zero, one, 10 two, three, four or five, was purged with dry nitrogen for 2 hours at room temperature to remove any physically adsorbed Si_2H_6 .

EXAMPLE 2: Preparation of B_xH_y Functionalized Aluminum Scavenger by B_2H_6
Deposition

15 60 cc of alumina with a specific surface area of 300 square meters per gram was loaded into a cylindrical reactor having a diameter of 2 cm and a length of 30 cm. The bed height formed by the alumina was 20 cm. The alumina was then purged with dry nitrogen for a period of 3 hours at a bed temperature of 150 degrees Celsius. A gas stream of dry nitrogen containing 10% B_2H_6 was passed through the reactor and alumina bed at a flow rate 20 of 1 liter per minute at a bed temperature of 20 degrees Celsius for 5 hours. The alumina, now supporting an active agent of the form B_xH_y , wherein x is one or two and y is zero, one, two, three, four or five, was purged with dry nitrogen for 5 hours at room temperature to remove any physically adsorbed B_2H_6 .

25 EXAMPLE 3: Preparation of P_xH_y Functionalized Aluminum Scavenger by PH_3
Deposition

30 60 cc of alumina with a specific surface area of 300 square meters per gram was loaded into a cylindrical reactor having a diameter of 2 cm and a length of 30 cm. The bed height formed by the alumina was 20 cm. The alumina was then purged with dry nitrogen for a period of 3 hours at a bed temperature of 150 degrees Celsius. A gas stream of dry nitrogen containing 15% PH_3 was passed through the reactor and inorganic bed at a flow rate of 1 liter per minute at a bed temperature of 300 degrees Celsius for 10 hours. The alumina, now supporting an active agent of the form P_xH_y , wherein x is one and y is two, was

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purged with dry nitrogen for 5 hours at room temperature to remove any physically adsorbed PH_3 .

EXAMPLE 4: Removal of Water and Oxygen Using Si_xH_y , B_xH_y , and P_xH_y ,
Scavengers

Table 1 shows the results of the use of the scavengers made in Examples 1-3 above for reducing the concentration of water and oxygen in a gas stream. These tests were conducted by placing each scavenger in a reactor in a fixed bed arrangement and introducing a nitrogen gas stream containing 10 ppm water (generated by diffusion vial) and 10 ppm oxygen (supplied from a calibrated gas) at a rate of 1 liter per minute. Water and oxygen concentrations in the exit gas were measured independently using Fourier Transformation Infrared (FTIR) spectroscopy and a total oxygen analyzer, respectively. As shown, all of the scavengers showed significant removal of water and oxygen.

Table 1.

Scavenger	Efficiency for moisture (ppb)	Efficiency for oxygen (ppb)
$\text{Si}_x\text{H}_y/\text{silica}$	<10	<1
$\text{B}_x\text{H}_y/\text{alumina}$	<10	<1
$\text{P}_x\text{H}_y/\text{alumina}$	<10	<1

Therefore, the scavenger, and its methods of making and using, according to the present invention are useful in producing a purified gas stream suitable for use, for example, in semiconductor manufacturing. Furthermore, the method for using the scavenger avoids the introduction of hydrocarbons and metals into the purified gas.

Although certain preferred embodiments have been described, it should be appreciated that variations, modifications and other embodiments of the present invention are possible, and all of these are to be regarded as being within the scope of the present invention.

What is claimed is:

1. A scavenger for reducing the concentration of an impurity in a gas,
comprising:
5 an inorganic support having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is one, two or three, and y is zero, one, two, three, four, five or six.
2. The scavenger of Claim 1 wherein said inorganic support is selected from the
10 group consisting of zeolites, alumina and porous silica.
3. The scavenger of Claim 1 wherein said inorganic support has a surface area of approximately 100 to 900 square meters per gram.
- 15 4. The scavenger of Claim 1 wherein said inorganic support has a pore size of approximately 3 to 1000 angstroms.
5. The scavenger of Claim 1 wherein said inorganic support is thermally stable at a temperature of approximately 20 to 300 degrees Celsius.
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6. The scavenger of Claim 1 wherein said active agent is selected from the group consisting of B_xH_y wherein x is one or two and y is zero, one, two, three, four or five; Si_xH_y wherein x is one or two and y is zero, one, two, three, four or five; AsH_y wherein y is zero, one, two or three; and PH_y wherein y is zero, one, two or three.
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7. The scavenger of Claim 1 wherein said active agent is SiH_y wherein y is zero, one, two or three.
8. The scavenger of Claim 1 wherein the concentration of said active agent is
30 approximately 3×10^{-5} to 1.5 moles per gram of said inorganic support.
9. A scavenger for reducing the concentration of an impurity in a gas,
comprising:
an inorganic support having disposed thereon an active agent selected from
35 the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y , and mixtures thereof, wherein x is

one, two or three, and y is zero, one, two, three, four, five or six, and disposed on said inorganic support, and wherein the concentration of said active agent is greater than approximately 5×10^{-4} moles per gram of said inorganic support.

- 5 10. A scavenger for reducing the concentration of an impurity in a gas, comprising:
- an inorganic support; and
- an active agent disposed on said inorganic support by contacting said inorganic support with a hydride gas selected from the group consisting of B_2H_6 , SiH_4 ,
10 Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof.
11. The scavenger of Claim 10 wherein said hydride gas contacts said inorganic support at a temperature of approximately 20 to 500 degrees Celsius.
- 15 12. The scavenger of Claim 10 wherein said active agent is made by contacting a mixture of said hydride gas and an inert gas with said inorganic support.
13. A method for making a scavenger for reducing the concentration of an impurity in a gas, comprising the step of:
- 20 contacting a hydride gas selected from the group consisting of B_2H_6 , SiH_4 , Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof, with an inorganic support.
14. The method of Claim 13 further comprising the step of heating said inorganic support in a dry environment prior to said contacting step.
- 25 15. The method of Claim 13 wherein said contacting step includes the step of contacting a mixture of said hydride gas and an inert gas with said inorganic support.
16. The method of Claim 13 wherein said inorganic support is selected from the
30 group consisting of zeolites, alumina and silica.
17. The method of Claim 13 wherein said inorganic support has a surface area of approximately 100 to 900 square meters per gram.

18. The method of Claim 13 wherein said inorganic support has a pore size of approximately 3 to 1000 angstroms.

19. The method of Claim 13 wherein said inorganic support is thermally stable at
5 a temperature of approximately 20 to 300 degrees Celsius.

20. The method of Claim 13 wherein said contacting step is conducted at a temperature of approximately 20 to 500 degrees Celsius.

10 21. The method of Claim 13 wherein said contacting step forms said active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is one, two or three, and y is zero, one, two, three, four, five or six.

22. The method of Claim 21 wherein said contacting step forms said active agent
15 selected from the group consisting of B_xH_y wherein x is one or two and y is zero, one, two, three, four or five; Si_xH_y wherein x is one or two and y is zero, one, two, three, four or five; AsH_y where in y is zero, one, two or three; and PH_y , wherein y is zero, one, two or three.

23. The method of Claim 22 wherein said active agent is SiH_y wherein y is zero,
20 one, two or three.

24. The method of Claim 13 wherein said contacting step is continued until said concentration of said active agent is approximately 3×10^{-5} to 1.5 moles per gram of said inorganic support.

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25. The method of Claim 13 further comprising the step of purging said inorganic support with an inert gas after said contacting step.

26. A method for making a scavenger for reducing the concentration of an
30 impurity in a gas, comprising the steps of:

contacting a hydride gas selected from the group consisting of B_2H_6 , SiH_4 , Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof, with an inorganic support;

pyrolyzing said hydride gas adsorbed on said inorganic support to form an active agent disposed on said inorganic support at a concentration greater than
35 approximately 5×10^{-4} moles per gram of said inorganic support.

27. A method for reducing the concentration of an impurity in a gas, comprising the step of:

contacting a gas containing an impurity with an inorganic support having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is an integer selected from the group consisting of one, two and three, and y is an integer selected from the group consisting of one, two, three, four, five and six.

28. The method of Claim 27 wherein said gas is selected from the group consisting of inert gases and hydride gases.

29. The method of Claim 27 wherein said gas is selected from the group consisting of hydrogen, nitrogen, helium, neon, argon, krypton, xenon, diborane, arsine, phosphine, silane, germane, hydrogen selenide, hydrogen telluride and ammonia.

30. The method of Claim 27 wherein said impurity is selected from the group consisting of oxidants and Lewis acids.

31. The method of Claim 30 wherein said impurity is selected from the group consisting of CO_2 , O_2 and H_2O .

32. The method of Claim 27 wherein said inorganic support is selected from the group consisting of zeolites, alumina and silica gel.

33. The method of Claim 27 wherein said inorganic support has a surface area of approximately 100 to 900 square meters per gram.

34. The method of Claim 27 wherein said inorganic support has a pore size of approximately 3 to 1000 angstroms.

35. The method of Claim 27 wherein said inorganic support is thermally stable at a temperature of approximately 20 to 300 degrees Celsius.

36. The method of Claim 27 wherein said active agent is selected from the group consisting of B_xH_y wherein x is one or two and y is zero, one, two, three, four or five; Si_xH_y wherein x is one or two and y is zero, one, two, three, four or five; AsH_y wherein y is zero, one, two or three; and PH_y wherein y is zero, one, two or three.

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37. The method of Claim 36 wherein said active agent is SiH_y wherein y is zero, one, two or three.

38. The method of Claim 27 wherein the concentration of said active agent is
10 approximately 3×10^{-5} to 1.5 moles per gram of said inorganic support.

39. A method for reducing the concentration of an impurity in a gas, comprising the step of:

15 contacting a gas containing an impurity with an inorganic support having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof, wherein x is an integer selected from the group consisting of one, two and three, and y is an integer selected from the group consisting of one, two, three, four, five and six, and wherein the concentration of the active agent is greater than approximately 0.0005 moles per gram of inorganic support.

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40. An apparatus for making a scavenger for reducing the concentration of an impurity in a gas, comprising:

a reactor having an inlet port, an outlet port and a bed for holding an inorganic support;

25 an inlet gas line connected to said inlet port for supplying a hydride gas selected from the group consisting of B_2H_6 , SiH_4 , Si_2H_6 , AsH_3 , PH_3 , GeH_4 and mixtures thereof to said reactor; and

a heater for supplying heat to said reactor.

30 41. An apparatus for reducing the concentration of an impurity in a gas, comprising:

a reactor having an inlet port, an outlet port and a bed for holding an inorganic support having disposed thereon an active agent selected from the group consisting of B_xH_y , Si_xH_y , As_xH_y , P_xH_y , Ge_xH_y and mixtures thereof wherein x is an integer

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selected from the group consisting of one, two and three, and y is an integer selected from the group consisting of one, two, three, four, five and six; and

an inlet gas line connected to said inlet port for supplying a gas containing an impurity to said reactor.

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FIG. 1

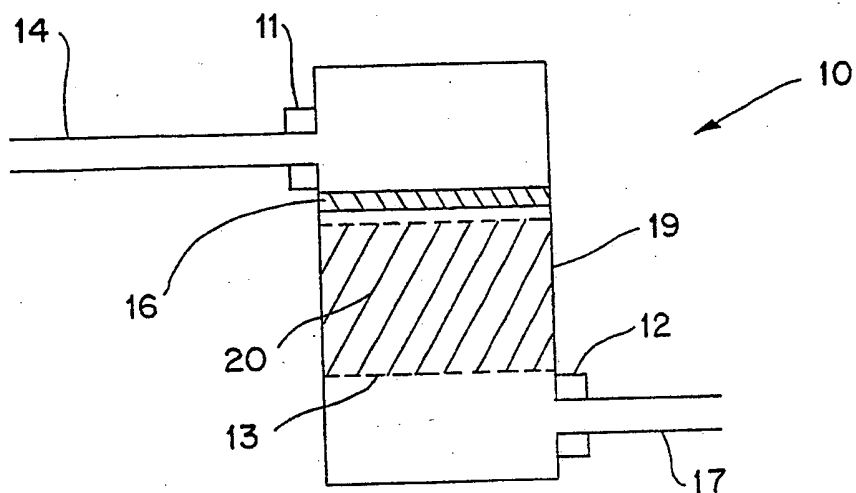
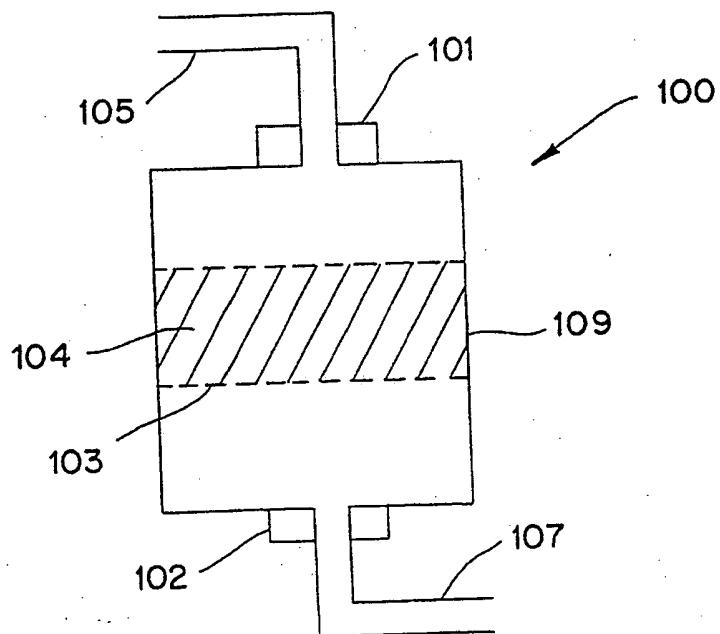


FIG. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/24971

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : B01D 53/28, 53/02; B01J 20/08, 20/18, 20/02

US CL : 502/400, 407, 64, 415; 423/219, 230, 210

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 502/400, 407, 64, 415; 423/219, 230, 210

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,565,677 A (YUSA) 21 January 1986, see col. 3, line 60 to col. 6, line 60.	1-41
X	US 4,683,125 A (YUSA) 28 July 1987, see col. 3, line 10 - col. 4, line 46.	1-41
A	US 4,720,444 A (CHEN) 19 January 1988, see entire document.	1-41
A	US 5,472,787 A (JOHNSON et al.) 05 December 1995, see entire document.	1-41
A	US 4,353,788 A (JEFFREY, et al) 12 October 1982, see entire document.	1-41

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 14 DECEMBER 1999	Date of mailing of the international search report 10 FEB 2000
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer STEVEN GRIFFIN Telephone No. (703) 308-0680 DEBORAH THOMAS PARALEGAL SPECIALIST

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